



# **Thermal Coating for Insensitive-Munitions Technologies**

**by Pauline Smith**

**ARL-TR-4317**

**November 2007**

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**Pauline Smith**

**Weapons and Materials Research Directorate, ARL**

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## 1. Objective

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The primary objective of this project is to improve the insensitive munitions (IM) performance of munitions by increasing the time before a slow cookoff reaction and reducing the hazard to no more than a type V (burning) reaction. This will be accomplished using state-of-the-art intumescent coatings.

This research will provide critical technology to mitigate collateral damage in the theatre of operations by ensuring safer transportation and storage of munitions in the field. Using coating technology, munitions response from environmental conditions and fragmenting warheads will be improved. To accomplish these objectives, coatings will be designed to provide technology that will offer increased time before initiating a slow cookoff reaction and to enable a controlled burn, whereby preventing violent reaction.

Recent advances in coating technologies offer the opportunity to protect munitions from cookoff events. This program will evaluate current packaging and platform coatings. In the first phase, the U.S. Army Research Laboratory (ARL) proposes to formulate a packaging intumescent coating that will provide improved thermal protection and potentially reduce blast performance or mitigate explosive reactions. The second phase will focus on formulating a missile-body coating that will offer improved thermal protection without impairing the aerodynamics of the missile. Finally, the third and highest risk phase will investigate the feasibility of developing a heat-reflective coating that could be applied and still meet the dimensional constraint of gun-launched munitions.

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## 2. Background

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Improving IM performance is a difficult task, especially with the slow cookoff reaction hazard. Retrofitting or block changing the currently several hundred older munitions in inventory unable to meet the current IM requirements is not feasible. It is especially difficult to qualify a new energetic material as a replacement fill for these systems. The best short-term method of mitigating these threats is through new concepts in thermal protection.

Several improvements in packaging have been investigated, including installing a fire blanket for insulation, insulative cradling of missiles, and venting rocket containers; however, each of these is munitions-specific and all have limitations in performance. A more universal solution is needed that has broad applicability and is relatively easy to implement in the short-term.

Additionally, for larger rocket motors, slow cookoff is a big problem because of the way the propellant detonates at elevated temperatures instead of burning. The goal is to lower the ignition temperature to lower the hazard to a burn.

Currently, the U.S. Army is evaluating coatings for ammunition containers to protect against IM hazards. An intumescent coating was tested that allowed packaged 60-mm mortar cartridges to successfully pass fast cookoff requirements in accordance with MIL-STD-2105C (1).

Intumescent fire-resistive coatings typically expand ~5–30 times during a fire testing. This expansion insulates the substrate from heat. This insulation will allow slow heating of the munitions, thereby allowing it to burn instead of detonate.

Intumescent coatings successfully protected against fast cookoff; however, several tests related to robustness of the intumescent coating system have shown poor results. This is mainly due to poor flexibility, impact, and marginal moisture resistance. They also do not fully meet sequential rough handling (SRH) requirements during ambient testing and especially at cold temperature extremes (the coatings tend to shatter off of the substrate). SRH tests are intended to be arduous to determine how long a system will endure extremes in the field before beginning to fail.

Risks associated with using intumescent coatings are that they will not provide enough insulation or that, by allowing slower heating, pockets of energetic material in the munitions may heat up, causing premature detonation. Testing is being conducted to verify that the applied coating thickness provides adequate thermal protection during fast and slow cookoff scenarios. Some coatings have been observed to have durability issues when exposed to harsh exterior environments and fire events. Testing is being performed to demonstrate coating performance over the possible environmental use conditions, which includes free-fall testing (at 65, 72, and 160 °F), high-humidity testing, and fire-exposure testing over a possible range of conditions.

An intumescent coating system for fire and heat protection is specified for 60-mm mortar containers. The projected intumescent coating system for ammunition containers has continuous field failures and has performed unsuccessfully during testing at the Aberdeen Test Center. The complexity of this project included paint delamination, cracking and chipping after the drop, and an impact-resistant test. The specified intumescent system has shown poor flexibility, impact, and marginal moisture resistance. Alternative coatings have shown progress in controlling the rate of heating during testing. Failures due to paint chipping, cracking, or water damage will most likely occur during rough handling with acrylic-based coatings. The alternatives, polyureas and epoxy-based coatings, performed exceptionally well during performance testing. Intumescent coatings that formed dense, firm char were observed to perform exceptionally well during cone calorimeter and high-velocity flame impingement testing.

The cone calorimeter is a fire testing instrument that quantitatively measures the inherent flammability of material through the use of oxygen consumption calorimetry. It is a standard technique under ASTM E 1354 and ISO 5660 (2, 3). This was designed primarily as a fire safety engineering tool, but has found great utility as a scientific tool providing an understanding of fire performance in relation to regulatory pass/fail tests. By looking at various parameters measured

by the cone calorimeter, one can correlate those measurements to other tests or understand why a material achieved a particular regulatory rating. Cone calorimeter is also sometimes used as part of regulatory schemes for high-fire endurance scenarios, such as MIL-STD-2031 (4) (U.S. Navy submarine applications), and some European mass transportation regulations (train, rail, and subway). Cone calorimeter testing was done on various coatings at the maximum possible heat flux ( $100 \text{ kW/m}^2$ ) to quantify the ignitability, heat release rate of the coatings, and heat transmission performance. This high heat flux represents severe fire conditions by local fires directly adjacent to a storage array. Several of the coatings tested had low heat release rate and smoke production while still providing excellent thermal protection. The impressive performance at  $100 \text{ kW/m}^2$  heat flux suggests that the chosen materials would do well in strict aviation, mass transport, and maritime applications.

Testing has also been performed on coatings selected from the cone calorimeter testing using a high-velocity flame impinging onto a coated steel panel. In these tests, the heat flux at the center of the sample was  $200 \text{ kW/m}^2$  ( $1100^\circ\text{C}$ ) after 5 min and remained at this level until the end of the test. This test is often used to mimic fire conditions produced by flammable liquid fuel pool fires and jet-fire scenarios. Two intumescent coatings performed exceptionally well in these tests, preventing temperatures on the unexposed side of the sample from exceeding  $350^\circ\text{C}$  for 10–15 min.

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### **3. Basic Technical Approach**

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This program will evaluate current packaging and platform coatings. In the first phase, ARL proposed to develop intumescent packaging coatings that would provide improved thermal protection and, potentially, blast performance. This effort consisted of market technology surveys to identify alternatives, evaluate, and down-select coatings. Based on data analysis, requirements development, and laboratory testing, the coatings will be further selected for additional testing. The initial laboratory testing will be conducted using coated cold-rolled steel panels for mechanical properties. Subsequently, IM tests will be conducted for alternative coatings to improve munitions response to those threats.

The second phase will focus on formulating a missile body coating that will offer improved thermal protection without impairing the aerodynamics of the missile. Finally, the third and highest risk phase will investigate the feasibility of developing a heat-reflective coating that could be applied and still meet the dimensional constraint of gun-launched munitions.

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## **4. Description of Proposed Work**

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Material testing is the process of evaluating materials for characteristics that include, but are not limited to, durability and overall strength. This is accomplished by placing them through arduous tests to see how long the system endures before it breaks down. In essence, any coating is reliably expected to stand up to the wear and tear of its target application.

To determine system failure analysis and performance improvement, the coating application process was evaluated to develop feasible production process parameter guidelines while optimizing curing time and temperature. For this project, laboratory tests were used to characterize and identify the best performers. Testing involved coating stress tests to establish base line data and to address and quantify adhesion and cracking problems using environmental stress, including accelerated weathering, impact, flexibility, and adhesion.

The coatings were down-selected and further tested for flame resistance, cone calorimeter, ballistic, fragment impact, bullet impact, slow cookoff, burn through, furnace, and drop.

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## **5. Technical Risk**

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Technical risks are demonstrating an alternative viable replacement that provides IM protection without significantly impacting performance. Overall, risks associated with using intumescent coatings are that they will not provide enough insulation or that, by allowing slower heating, pockets of energetic material in the munitions may heat up causing premature detonation.

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## **6. Testing Overview**

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The U.S. Army is evaluating coatings for the outside of munitions containers. Using coating technology, the munitions' response to environmental conditions and fragmenting warheads will be improved. Before being used in the field, munitions containers coated on the outside must be exposed to a large-scale fire and the cookoff time must be recorded. Initial screening of coatings is being performed through a series of small-scale fire tests using the ASTM E 1354 cone calorimeter. Data were taken to compare the fire and insulating performances of the coating samples.

Eleven coatings were evaluated in this test series. Coating samples received were identified as follows: 05-118-78A, 05-118-78B, 05-118-78C, LS-RF-150, LS-RF-180, N-8445A/H<sub>2</sub>O<sub>2</sub>B, RH-30IR, Intumet 2002, AlbiClad 800, Chartek 1709, and Nullifire System E. Recommendations on the coatings to be included in the next series of testing are also provided at the end of this report.

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## 7. Experimental

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### 7.1 Standard ASTM E 1354 Cone Calorimeter Tests

ASTM E 1354 provides a small-scale test procedure to measure the ignitability, heat release rate, mass loss rate, and combustion product generation rate of a material exposed to a specified irradiance level. During a test, a 100- × 100-mm sample is placed beneath the conical-shaped heater that provides a uniform irradiance on the sample surface (see figure 1). The sample mass is constantly monitored using a load cell, and the effluent from the sample is collected in the exhaust hood above the heater. In the duct downstream of the hood, the flow rate, smoke obscuration, and O<sub>2</sub>, CO<sub>2</sub>, and CO concentrations are continuously measured.

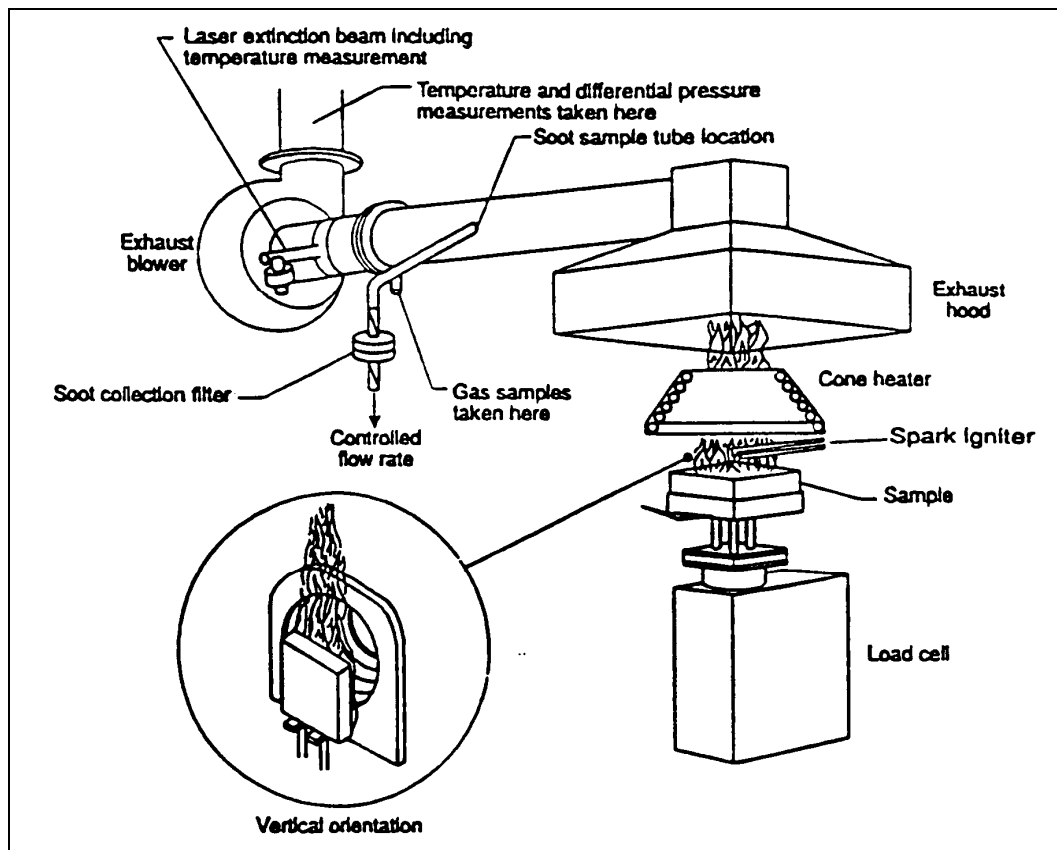


Figure 1. ASTM E 1354 cone calorimeter test apparatus.

A spark igniter 12.5 mm above the sample surface is used to initiate burning any combustible gas mixture produced by the sample. Once the sample ignites, burning the sample causes a reduction in the oxygen concentration within the effluent collected by the hood. This reduction in oxygen concentration has been shown to correlate with the heat release rate of the material, 13.1 MJ/kg of O<sub>2</sub> consumed. This is known as the oxygen consumption principle. Using this principle, the heat release rate per unit area of the sample is determined with time, using measurements made in the duct.

The ASTM E 1354 standard requires the following data be reported for each material tested:

- Time to ignition (seconds).
- Peak heat release rate (kilowatts per square meters).
- Heat release rates averaged over various time periods, starting with the time of ignition (kilowatts per square meters).
- Effective heat of combustion (megajoules per kilograms).
- Mass loss rate per unit area (kilograms per seconds square meters).
- Percent specimen mass loss (percent).
- Average smoke specific extinction area (square meters per kilograms). Smoke production from a material has the rational units of square meters, representing the extinction cross section of the smoke. This is normalized by the amount of specimen mass loss (kilograms).
- Average CO and CO<sub>2</sub> production yields (kilograms per kilograms).

In addition, the standard measurements and the temperature on the unexposed side of the sample were measured at the middle of the sample using a glass braid, bare bead, type K thermocouple. Digital photographs were taken of the material before and after fire testing.

## **7.2 Test Samples**

Test samples were ~100 × 100 mm. All samples tested were composed of a sheet-steel backing with a coating on one side, except for samples N-8445A/H<sub>2</sub>O<sub>2</sub>OB and RH-30IR, which did not utilize a steel backing material. The steel substrate had an ~1-mm thickness. Table 1 gives a description of each sample, and figure 2 shows photographs of each sample. Coating thickness was determined by an average of four thickness measurements obtained using digital calipers. The mass of the coating per unit area was determined by subtracting the sample weight from the weight of the steel plate.



Table 1. Description of coatings tested.

No.	Description	Specimen ID	Description	Average Coating Thickness (mm)	Coating Mass Per Unit Area (kg/m <sup>2</sup> )
1	Intumet 2002 (118 mil)	Intumet_2002	Brown dull finish, smooth coating	3.00	3.92
2	Pitt char XP (98–138 mil) Nonflame retardant polyurea (30 mil)	05-118-78A	Glossy black, smooth coating	3.00	4.24
3	Pitt char XP (138–177 mil) Nonflame retardant polyurea (30 mil)	05-118-78B	Glossy black, smooth coating	4.00	5.58
4	Pitt char XP (98–138 mil) Flame retardant polyurea (30 mil)	05-118-78C	Glossy black, smooth coating	3.00	4.52
5	Albi 487S primer AlbiClad 800 coating (115–125 mil)	AlbiClad 800	White dull finish, uneven coating	3.18	4.02
6	Carbogard 890 primer Chartek 1709 coating (115–125 mil)	Chartek 1709	Grey dull finish, uneven coating	3.18	3.14
7	Intergard 251 primer Nullifire System E coating (115–125 mil)	Nullifire System E	Green dull finish, uneven coating	3.18	3.30
8	LS-RF-150	LS-RF-150	Glossy black coating with silver speckle	3.20	4.27
9	Flame-retardant epoxy primer N-8445A/H2OB olive drab MIL-DTL-64159 type II	N-8445A/H2OB	Green dull finish, smooth coating	4.08	5.47
10	LS-RF-180	LS-RF-180	Glossy black coating with silver speckle	4.45	5.09
11	Flame-retardant epoxy primer RH-30IR higher IR flame retardant latex emulsion	RH-30IR	Green dull finish, smooth coating	3.88	5.62

### 7.3 Test Conditions

All tests were conducted in the horizontal configuration using an  $\sim 95\text{-kW/m}^2$  exposure heat flux. Samples 1–4 and 8–11 were tested once at the exposure heat flux, while samples 5–7 were tested in duplicate.

One of the more important measurements in these tests was the unexposed side temperature of the steel plate. Sample holders recommended in ASTM E 1354 are constructed of steel. These steel sample holders may serve as a path to conduct heat prematurely to the unexposed side of

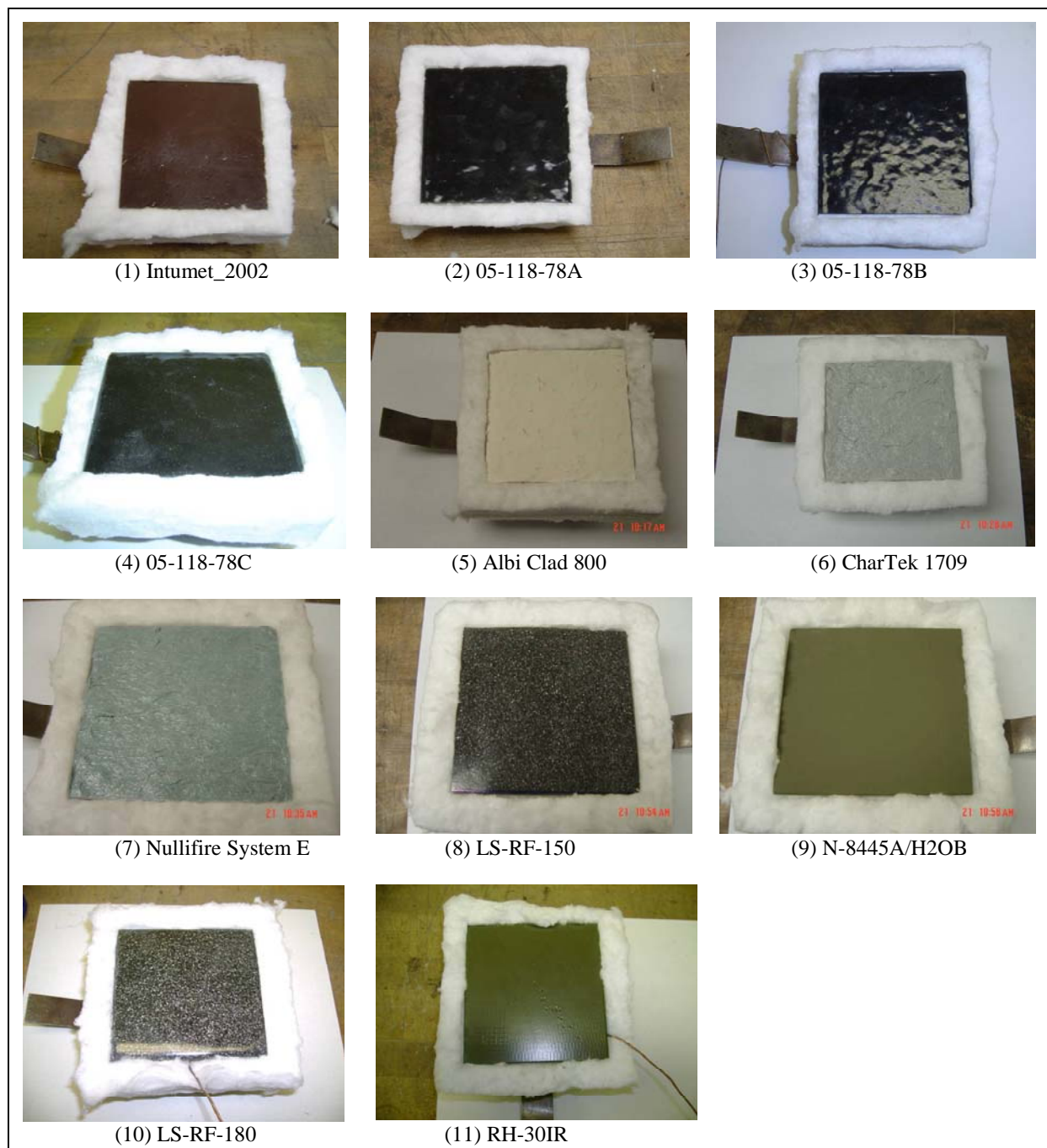


Figure 2. Pictures of coating samples tested.

the sample. To reduce conduction effects to the unexposed side, samples were tested on a ceramic insulation holder, made using Unifrax 96-kg/m<sup>3</sup> density ceramic insulation. The ceramic insulation was 125 × 125 mm, 25 mm thick, with a 100- × 100-mm, ~3-mm-deep cutout in the center of the insulation. The sample was placed into the cutout so that the sample surface was flush with the ceramic insulation surface. The thermocouple measuring the unexposed temperature of the sample was placed between the sample and ceramic insulation with the

thermocouple bead located at the middle of the sample. The insulation was placed on top of a standard test tray filled with 25-mm-thick ceramic insulation, making it easy to place the sample underneath the heater.

Samples were tested 37.5 mm below the heater. In accordance with the standard, samples are typically tested 25 mm below the heater. However, these samples were expected to expand which would increase the heat-flux incident on the surface of the char. The heat flux from the heater will increase by ~10% with a 25-mm increase in height. Therefore, samples were moved 12.5–37.5 mm below the heater to compensate for the increase in heat flux as the sample expands during the test.

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## 8. Test Results

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The fire performances of the different coatings were compared, based upon the coatings' ignitability, heat release rate, and heat of combustion. The standard results from the cone calorimeter testing are provided in appendix A.

Table 2 gives a summary of the cone calorimeter test results. All coatings were measured to ignite within the first 14 s of exposure, with the Nullifire System E samples having the most resistance to ignition. Coatings 05-118-78A, 05-118-78B, and 05-118-78C performed significantly worse than the other coatings with peak heat release rates ranging from 803 to 1052.3 kW/m<sup>2</sup>. Although the heat release rates measured for coatings N-8445A/H<sub>2</sub>O<sub>2</sub> and RH-30IR were nearly half that of the coatings previously mentioned, peak heat release rate values of 486 and 532.6 kW/m<sup>2</sup>, respectively, were still more than double that measured for the remaining six samples. In general, the remaining six coatings tested (i.e., Intumet 2002, Albi Clad 800, Chartek 1709, Nullifire System E, LS-RF-150, and LS-RF-180) had similar fire performance results, with the average heat of combustion values ranging from 12.2 to 25.1 MJ/kg, average heat release rates ranging from 77 to 164.5 kW/m<sup>2</sup>, and the average peak heat release rates ranging from 126 to 264 kW/m<sup>2</sup>. Figure 3 contains heat release rate curves from a single test on each coating. As indicated from the data in the summary table, the 05-118-78A, 05-118-78B, and 05-118-78C coatings were significantly worse than the other coatings. The measured burning durations for the coatings tested varied widely, ranging from 156 s (measured for N-8445A/H<sub>2</sub>O<sub>2</sub>) to 667 s (measured for LS-RF-150). With respect to material flammability, the best performing coatings in this test series were Chartek 1709, Albi Clad 800, and Nullifire System E.

Table 2. Summary of cone calorimeter test results on the coatings.

Specimen ID	Irradiance (kW/m <sup>2</sup> )	Initial Mass (g)	Percent Weight Loss (%)	Time to Ignition (s)	Burn Duration (s)	Test Average Eff. HOC (MJ/m <sup>2</sup> )	Total Head Released (MJ/m <sup>2</sup> )	Test Average HRR (kW/m <sup>2</sup> )	Peak HRR (kW/m <sup>2</sup> )	Time of Peak HRR (s)	Test Average Smoke SEA (m <sup>2</sup> /kg)	Test Average CO <sub>2</sub> Yield (kg/kg)	Test Average CO Yield (kg/kg)
Intumet_2002	95	39.2	51.3	6	258	20.3	40.9	156.6	264.0	85	808	1.472	0.027
05-118-78A	95	42.4	77.4	3	339	24.1	79.0	231.8	1052.3	27	529	1.846	0.026
05-118-78B	95	55.8	70.8	3	339	24.1	79.0	231.8	1052.3	27	529	1.846	0.026
05-118-78C	95	45.2	73.4	3	487	22.8	75.5	154.5	803.0	21	576	1.692	0.061
1-Albi Clad 800	95	38.4	58.9	2	237	13.3	30.0	126	267	15	211	0.968	0.033
2-Albi Clad 800	95	40.7	57.2	2	224	11.1	25.9	115	243	19	237	0.919	0.028
1 and 2 Albi Clad 800 average	95	39.6	58.1	2	231	12.2	28.0	120	255	17	224	0.943	0.030
1-Chartek 1709	95	29.7	65.0	9	468	17.7	34.2	73	143	19	821	1.442	0.104
2-Chartek 1709	95	32.8	62.8	8	468	18.3	37.8	80	109	11	628	1.450	0.095
1 and 2 Chartek 1709 average	95	31.3	63.9	9	468	18.00	36.0	77	126	15	725	1.446	0.099
1-Nullfire System E	95	33.0	71.5	14	341	19.3	45.5	132	241	13	1147	1.432	0.038
2-Nullfire System E	95	32.8	72.3	12	340	18.9	44.7	131	237	13	1123	1.440	0.035
1 and 2 Nullfire System E average	95	32.9	71.9	13	341	19.1	45.1	132	239	13	1135	1.436	0.037
LS-RF-150	95	44.0	78.0	5	667	24.9	85.5	127.8	210.6	14	302	2.109	0.044
N-8445A/H <sub>2</sub> OB	95	55.2	40.6	10	156	20.1	45.0	285.4	486.0	62	1098	1.547	0.050
LS-RF-180	95	52.0	66.0	5	635	25.1	104.8	164.5	250.3	221	347	2.075	0.031
RH-30IR	95	55.2	39.9	12	168	20.9	45.9	271.4	532.6	64	1214	1.597	0.055

Notes: Initial mass and percent weight loss were based on the mass of the coating only.

HOC = heat of combustion.

HRR = heat release rate.

SEA = specific extinction area.

Eff. = efficiency.

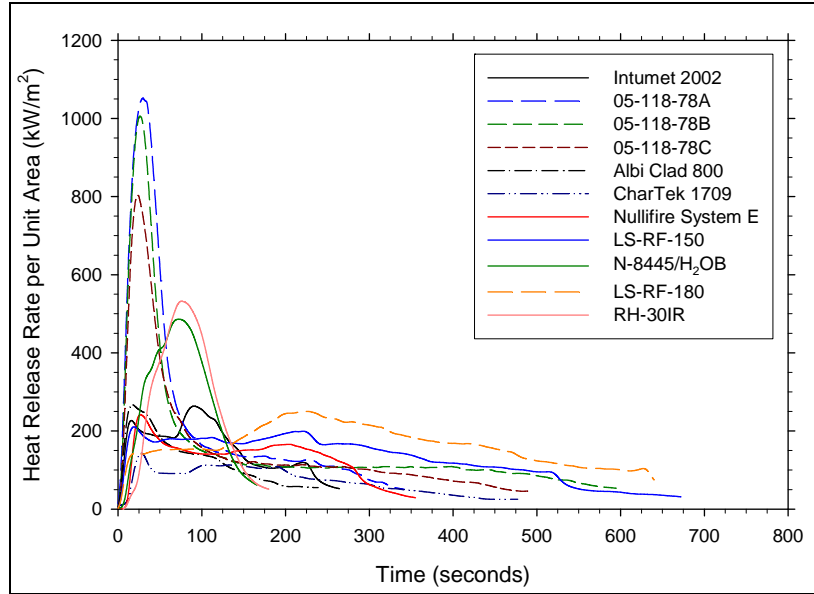


Figure 3. Heat release rates of the tested coatings.

Figure 4 gives the temperature on the unexposed side of the steel plate measured during the cone tests. Overall, LS-RF-180 provided the most thermal insulation, while N-8445A/H<sub>2</sub>O<sub>B</sub> provided the least protection to temperature rise on the unexposed side. The critical, unexposed-side temperature threshold for the testing was 350 °C. Table 3 summarizes the times required to reach the 350 °C threshold. Based upon these results, the best performing coatings, with respect to resistance to heat transmission, were LS-RF-150, LS-RF-180, and Chartek 1709, with Chartek 1709 being the only of the three coatings to have a nonfriable intumescent material.

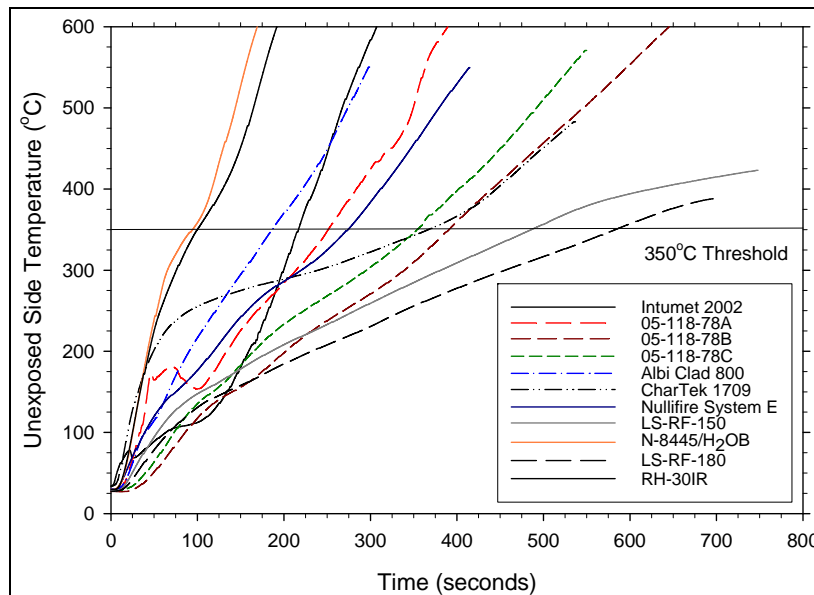


Figure 4. Unexposed-side temperature for different coating samples.

Table 3. Time to exceed 350 °C temperature threshold.

Specimen ID	Irradiance (kW/m <sup>2</sup> )	Time to Exceed 350 °C (s)
Intumet 2002	95	218
Intumet 2002 average	95	218
05-118-78A	95	252
05-118-78A average	95	252
05-118-78B	95	391
05-118-78B average	95	391
05-118-78C	95	355
05-118-78C average	95	355
1-Albi Clad 800	95	200
2-Albi Clad 800	95	209
1 and 2 Albi Clad 800 average	95	205
1-CharTek 1709	95	368
2-CharTek 1709	95	438
1 and 2 CharTek 1709 average	95	403
1-Nullifire System E	95	276
2-Nullifire System E	95	270
1 and 2 Nullifire System E average	95	273
LS-RF-150	95	487
LS-RF-150 average	95	487
N-8445A/H <sub>2</sub> OB	95	95
N-8445A/H <sub>2</sub> OB average	95	95
LS-RF-180	95	584
LS-RF-180 average	95	584
RH-30IR	95	100
RH-30IR average	95	100

Samples were inspected after the test to evaluate the state of the intumescent char. Figure 5 shows pictures of the samples after the test, and table 4 contains the char thickness measured using digital calipers after the test. The char observed following the exposure of coatings Intumet 2002, LS-RF-150, and LS-RF-180 was a light, friable char with average thicknesses of 9.5, 90, and 98 mm, respectively. The rest of the samples, other than N-8445A/H<sub>2</sub>OB and RH-30IR, had a rigid block of char remaining after exposure.

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## 9. Conclusions

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Cone calorimeter tests were conducted on 11 coating samples in the horizontal orientation with at a 95-kW/m<sup>2</sup> exposure heat flux. Data were taken to compare the insulating and fire performance of the coating samples. Plots of these data for the different coatings are provided in figures 6 and 7. For comparison purposes, results from a cone calorimeter test with 7-mm-thick pine are also provided in the figures. The 7-mm-thick pine has a 3.2-kg/m<sup>2</sup> mass per unit area,



Figure 5. Photographs of coatings after testing.

Table 4. Coating intumescent char thickness measured after test.

Specimen ID	Char Thickness (mm)
Intumet2002	9.5
05-118-78A	21
05-118-78B	32
05-118-78C	26
Albi Clad 800	7.2
CharTek 1709	27
Nullfire System E	12
LS-RF-150	90
N-8445/A/H2OB	—
LS-RF-180	98
RH-30IR	—

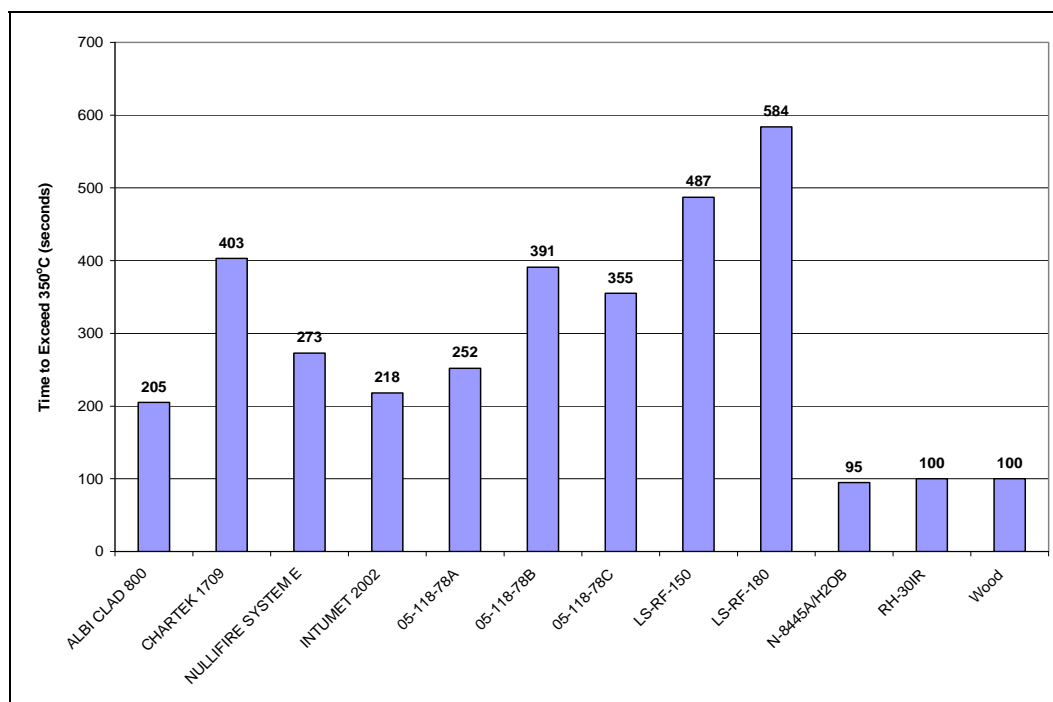


Figure 6. Comparison of time to exceed 350 °C on the unexposed side.

which is similar to the average mass per unit of the tested coatings. LS-RF-150 and LS-RF-180 had the best insulating performances of all coatings tested, resulting in the longest times to exceed the 350 °C temperature threshold (487 and 584 s, respectively). However, these coatings formed a light, friable char, and it is uncertain how they will perform when exposed to a large-scale pool fire where the higher-velocity environment may degrade the char more readily. All of the other coatings, except for Intumet 2002, formed a more rigid char, which is likely to be more resilient to conditions during a large-scale pool fire. Of these more durable coatings, the third



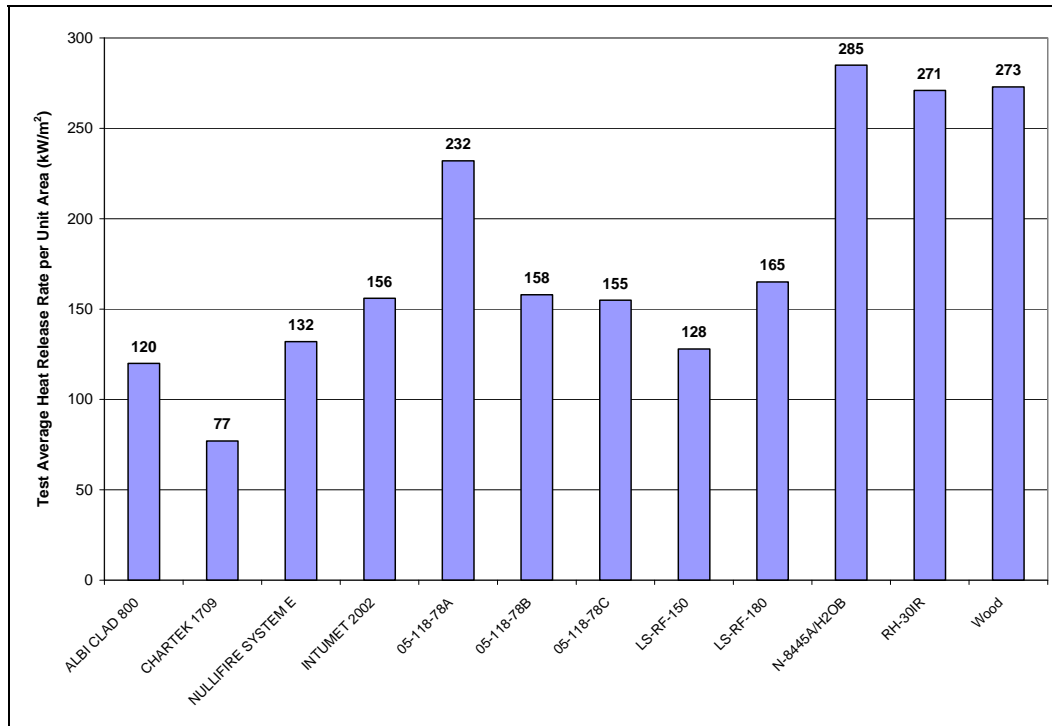


Figure 7. Comparison of test average heat release rates.

most thermally resistant coating was CharTek 1709, which lasted 403 s before exceeding the 350 °C threshold. This coating was also one of the top four performers, with respect to resistance to ignition, and, once ignited, resulted in the lowest average heat release rate of all coatings tested.

## 10. Recommendations

Twenty-one coatings were tested in three small-scale test series. The data resulting from these tests were evaluated based upon material flammability characteristics and the coatings' overall resistance to transmitting heat to the coating substrate. Based upon this evaluation, five coatings were identified for burn-through testing: LS-URF-140, LS-RF-180, Chartek 1709, Nullifire System E, and PittChar XP 2.65 mm. Coatings LS-URF-140 and LS-RF-180 were the most resistant to transmitting heat to the coating substrate; however, these coatings generate a light, friable char, which could degrade when exposed to a more turbulent fire environment. The remaining three coatings (Chartek 1709, Nullifire System E, and PittChar XP 2.65 mm) were all chosen for their above-average material flammability performance and resistance to heat transmission. The performances of the coatings just listed, with respect to all coatings tested, are illustrated in figures 8 and 9.

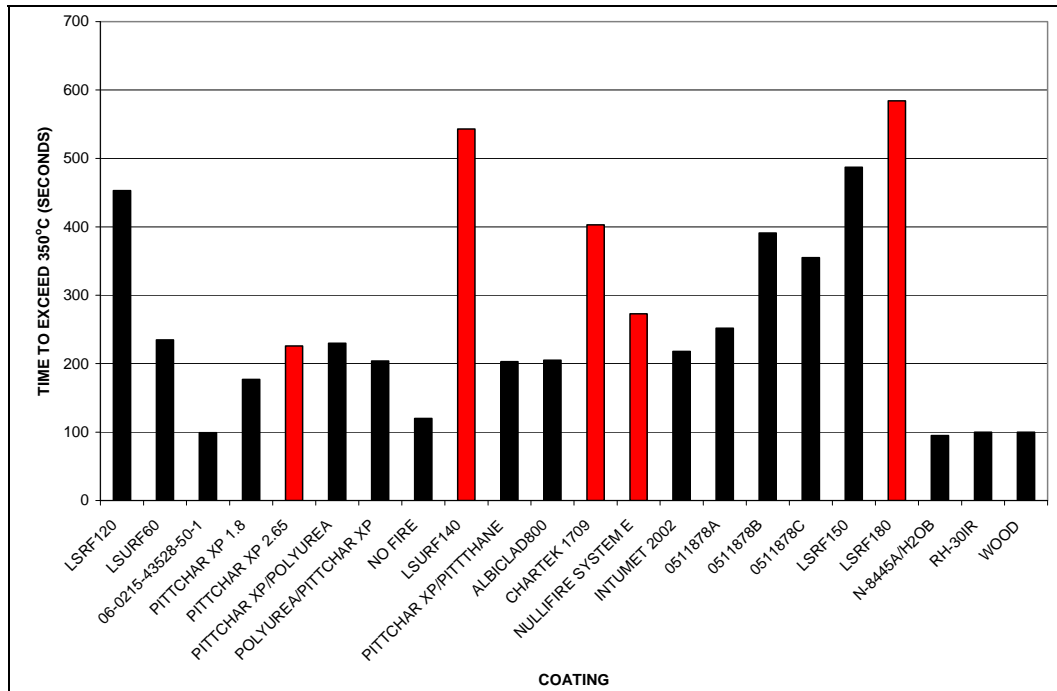


Figure 8. Comparison of time to exceed 350 °C on the unexposed side for all coatings tested. Coatings identified for burn-through testing are identified in red.

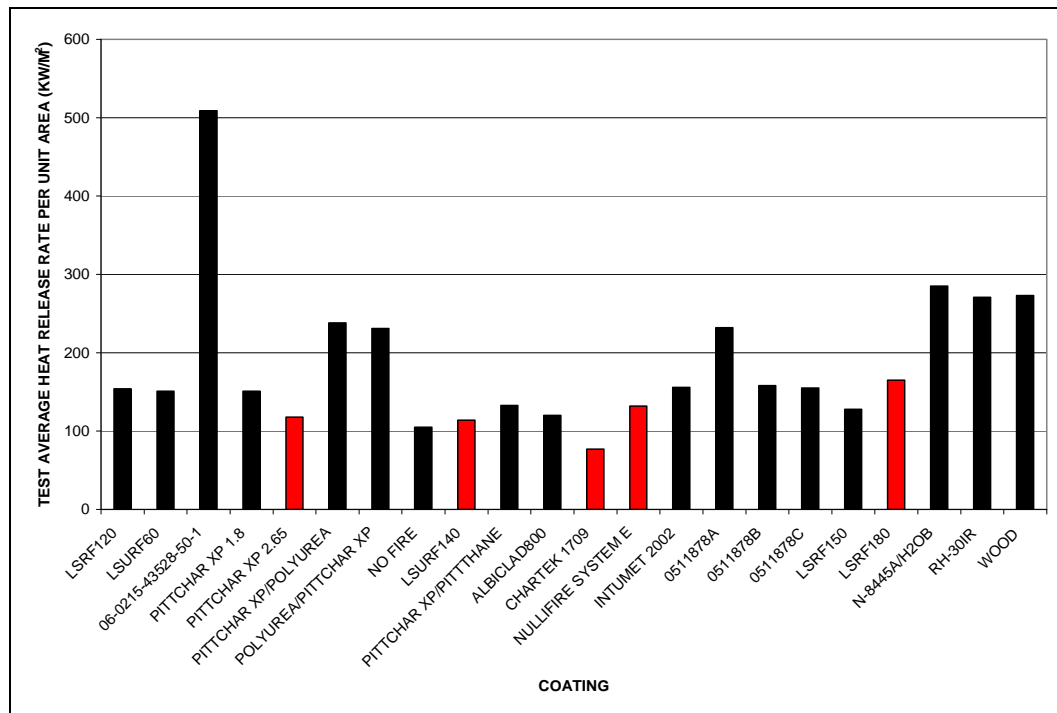


Figure 9. Comparison of test average heat release rates for all coatings tested. Coatings identified for burn-through testing are shown in red.

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## 11. Burn-Through Fire Testing of Coatings for Munition Containers

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### Testing Overview

Initial screening of coatings was performed through a series of small-scale fire tests using the ASTM E 1354 cone calorimeter. Based upon the data from these test series of 21 coatings, three of the top performing coatings were identified for further evaluation. The coatings identified as potential candidates for use on the outside of munitions containers were Nullifire System E, PittChar XP/Polyurea, and LS-URF-140. These coatings were chosen based on their insulating and fire performance when tested in the cone calorimeter.

Each of the coatings was applied to a steel substrate and subjected to burn-through testing conducted in accordance with MIL-STD-2031. The performance of each coating when subjected to burn-through fire testing is described in this report. Recommendations on the coatings to be included in the next series of testing are provided in section 14 (Conclusions) of this report.

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## 12. Experimental

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### 12.1 Burn-Through Test Apparatus

The apparatus used in this testing consisted of the test sample, a mounting frame, and a propane burner positioned perpendicular to the mounted sample. See figure 10 for an illustration of the experimental setup. The sample mounting frame was constructed so that the sample was mounted in a vertical orientation and was enclosed on all four sides. The test sample was enclosed in an effort to establish a more uniform heat-flux distribution across the exposed surface. Boundary conditions comparable to those adopted in previous small-scale testing were used in this test series. The walls enclosing the four sides of the test sample provided an enclosure depth of ~0.3 m (12 in) and were constructed from 12.7-mm (0.5-in) ceramic insulation board. Furthermore, because of the importance of the temperature measurements on the unexposed side of the steel substrate, this boundary was insulated using a ceramic blanket. A single layer of 0.5-m<sup>2</sup> (20-in), 25.4-mm (1-in)-thick Unifrax 96-kg/m<sup>3</sup> density ceramic insulation was installed on the steel surface.

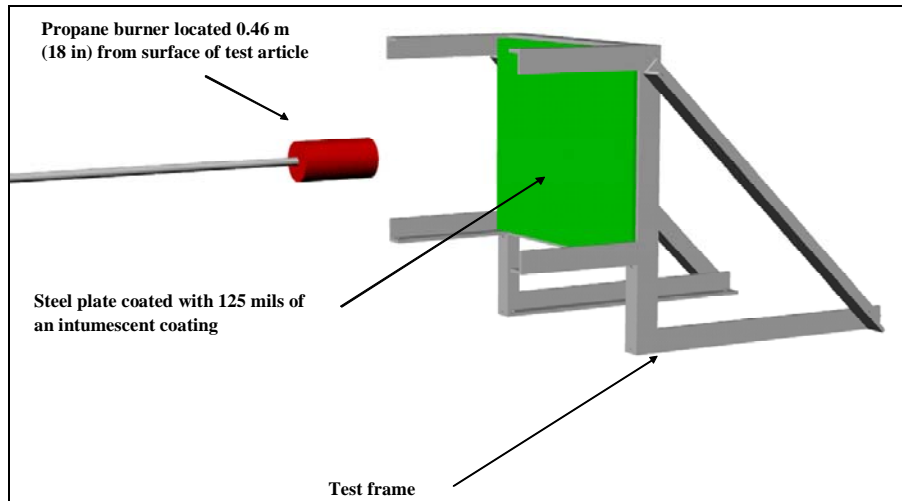


Figure 10. Overview of test apparatus.

As shown in figure 10, the propane burner was located 0.46 m (18 in) from the coated surface of the test sample and centered with respect to the sample surface. Gas flow to the burner was controlled so that an exposure representative of that expected from a UL 1709 fire exposure was achieved (5).

Temperature measurements were collected throughout all tests conducted at various locations on the test panel and test apparatus. Bare-bead, type K, 24-Ga thermocouples were used to measure temperature at the following locations during testing;

- One thermocouple located 50.8 mm (2 in) from the tip of the burner
- One thermocouple located on the exposed surface of the sample
- One thermocouple located 38.1 mm (1.5 in) from the unexposed side of the sample

In addition to these thermocouples, four temperature measurements were collected from the unexposed surface of each test panel. In these locations, the thermocouples were welded directly to the unexposed steel substrate onto which the coatings were applied. The location of these thermocouples, as specified in MIL-STD-2031, was as follows:

- One thermocouple located 25.4 mm (1 in) from the top edge of the test panel along the vertical centerline
- One thermocouple located in the center of the test panel
- One thermocouple located 25.4 mm (1 in) from the bottom edge of the test panel along the vertical centerline
- One thermocouple located 0.1 m (4 in) to the right of the previously mentioned lower thermocouple

These thermocouples were used to evaluate the transmission of the heat through the test sample when subjected to an exposure comparable to that obtained in an UL 1709 fire exposure. In addition to temperature measurements, digital photographs and video were taken throughout fire testing.

## 12.2 Test Samples

Test samples were  $0.46 \times 0.46$  m ( $18 \times 18$  in). All tested samples were composed of a sheet-steel backing with a coating applied to only one side. The steel substrate had an  $\sim 1$ -mm thickness. Table 5 gives a description of each sample, and figure 11 shows photographs of each sample. Coating thickness was determined by an average of four thickness measurements obtained using digital calipers. The mass of the coating per unit area was determined by subtracting the sample weight from the weight of the steel plate.

Table 5. Description of coatings tested.

No.	Description of Coating	Specimen ID	Average Coating Thickness (mm)	Coating Mass Per Unit Area ( $\text{kg/m}^2$ )
1	Intergard 251 primer Nullifire System E coating	Nullifire System E	3.71	3.58
2	LS-URF-140	LS-URF-140	3.65	3.06
3	Pitt char XP/polyurea	05-118-78B	4.15	3.97

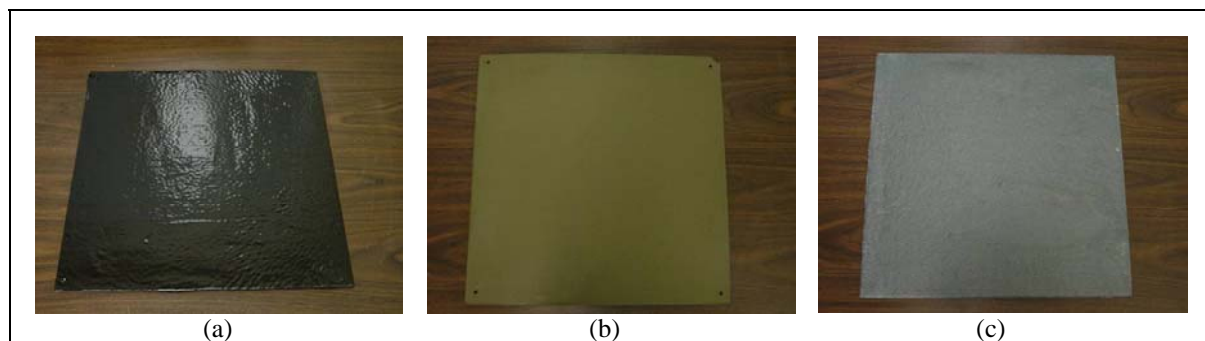


Figure 11. Photographs of test panel coated with (a) PittChar XP/Polyurea, (b) LS-URF-140, and (c) Nullifire System E.

## 12.3 Test Conditions

All tests were conducted with samples mounted in the vertical orientation using the exposure heat-flux profile provided in figure 12. All coatings were tested once at this exposure. A photograph of the exposure fire used to produce  $200\text{-kW/m}^2$  heat fluxes at the center of the test panels is provided in figure 13.

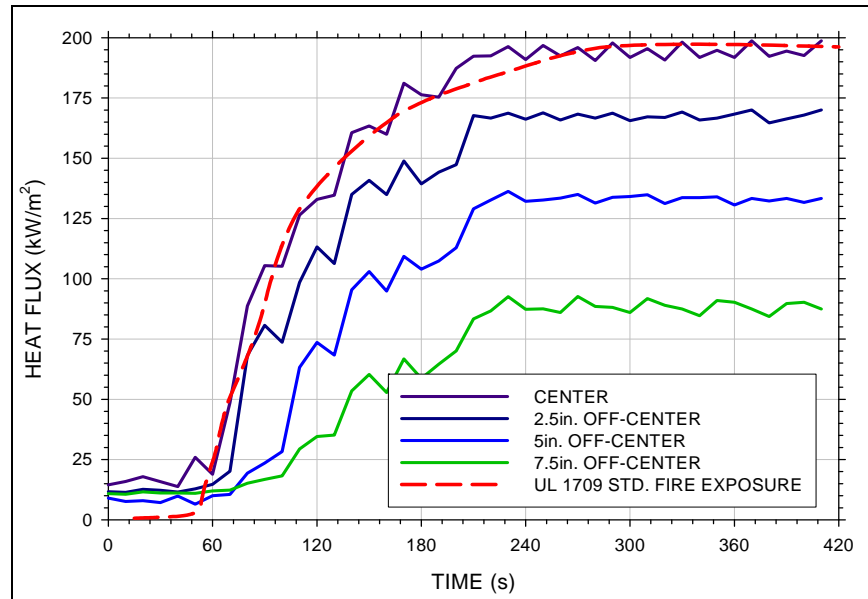


Figure 12. Heat-flux exposure used during testing of coated samples. Also shown in the plot are calculated heat fluxes resulting from a UL 1709 exposure fire.



Figure 13. Photograph of the propane burner exposure fire.

Once instrumented, the ceramic blanket insulation was installed on the backside of the panel and the panel was mounted within the test frame. After mounting the panel, the ceramic board enclosure walls were installed. The walls were butted up against the surface of the coatings; thus, the actual exposed surface area for each coating was 0.43 m<sup>2</sup> (17 in). Two min of background data were collected prior to igniting the propane burner. The propane burner remained on until all temperatures on the exposed surface of the test sample exceeded the temperature threshold of 350 °C at all measurement locations, at which point the burner was secured. Following each test, intumescent thicknesses were collected at five points on the surface of the sample. These thicknesses were measured obtained using digital calipers.

### 13. Test Results

The three intumescent coatings tested were evaluated based upon the insulating performance of the coating. Additional observations, with respect to coating ignitability and durability, are also included in these results. Table 6 summarizes the test results for all three coatings evaluated.

Table 6. Summary of results from burn-through testing.

Test No.	Coating ID	Time to Ignition (s)	Time to Intumescent Falling Off Surface (s)	Time to Exceed 350 °C Threshold (s)		Average Char Thickness (mm)
				First Thermocouple	All Thermocouples	
1	Nullifire System E	85	NA	458	700	18
2	LS-URF-140	45	120	326	447	23.7
3	PittChar XP	20	NA	483	900	213

Note: NA = not applicable.

The first panel was coated with 3.71 mm (146 mil) of the Nullifire System E coating. Ignition of this coating occurred 85 s after initiation of the burner. The 350 °C threshold was initially exceeded 458 s (7 min, 38 s) after initiation of the propane burner. Temperatures on the unexposed surface of this test panel are presented in figure 14.

As shown in figure 14, all backside temperatures exceeded the temperature threshold ~700 s (11 min, 40 s) after exposure to the burner. The Nullifire System E coating remained attached to the steel substrate throughout the fire exposure and after the panel was removed from the test frame. Posttest intumescent char thickness measurements resulted in an 18-mm average thickness. A photograph of the intumescent coating following the fire exposure is provided in figure 15.



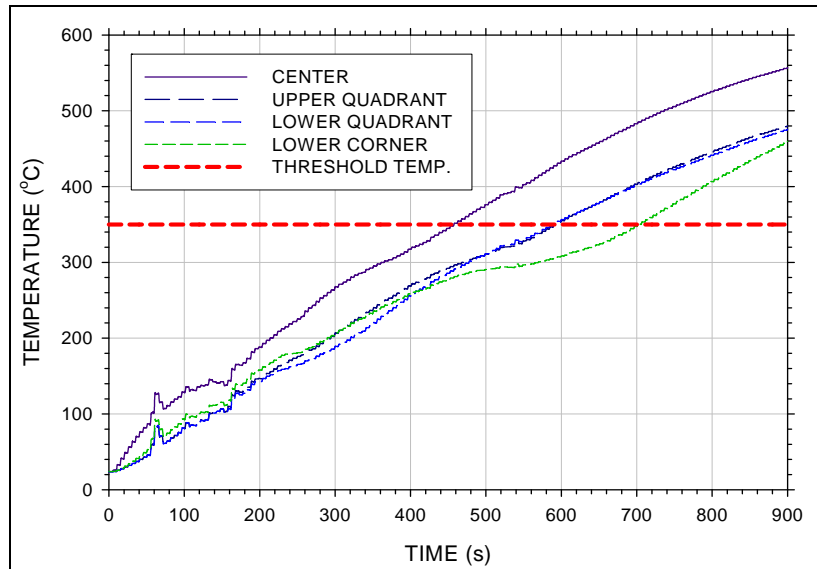


Figure 14. Unexposed surface temperatures measured during exposure of the Nullifire System E coating.



Figure 15. Photograph of the Nullifire System E coating after burn-through testing.



The second panel was coated with 3.65 mm (144 mil) of LS-URF-140. This coating was observed to ignite ~45 s after initiation of the burner. Early in the exposure of this panel (i.e., ~2 min after ignition), sections of intumesced material began falling from the test panel. It was also observed that when these sections fell from the sample, the intensity of the burning on the panel and smoke production of the panel fire would increase. In this test, the 350 °C threshold was initially exceeded 326 s (5 min, 26 s) after initiation of the propane burner. Temperatures on the unexposed surface of this test panel are presented in figure 16.

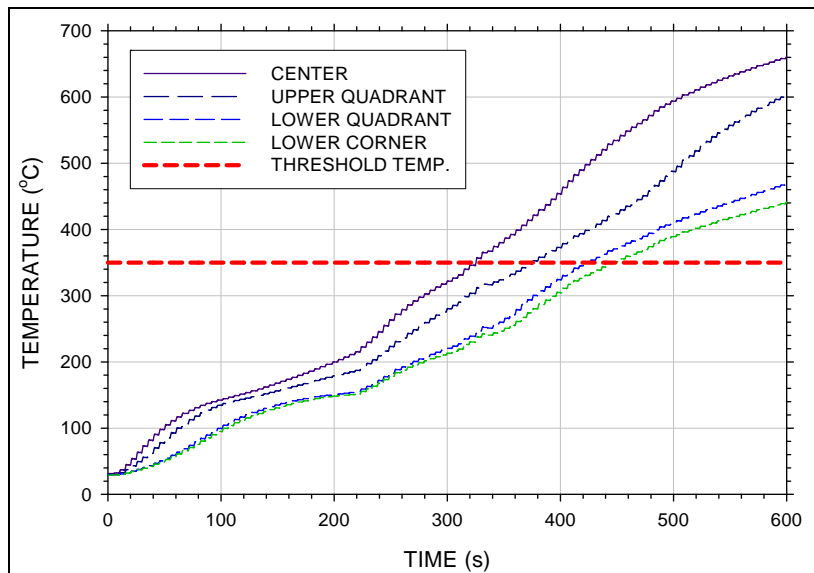


Figure 16. Unexposed surface temperatures measured during exposure of LS-URF-140.

As shown in figure 15, all backside temperatures exceeded the temperature threshold ~447 s (7 min, 27 s) after exposure to the burner. As stated earlier, the intumesced material resulting from this coating was unable to remain in place over the duration of the exposure. Sections of intumesced material were observed to fall from the test panel as early as 2 min after exposure and continued to fall from the test panel throughout the test. Posttest intumescent char thickness measurements resulted in a 23.7-mm average thickness. A photograph of the intumescent coating following the fire exposure is provided in figure 17. Also shown in figure 17 is a photograph of the test sample after being removed from the test frame. The photograph illustrates that all of the intumescent char fell off of the surface of the steel when the test panel was moved.

The third panel was coated with 4.15 mm (163 mil) of PittChar XP. This coating ignited 20 s after initiation of the burner. The 350 °C threshold was initially exceeded 483 s (8 min, 3 s) after initiation of the propane burner at the center of the panel. Temperatures on the unexposed surface of this test panel are presented in figure 18.



Figure 17. Photograph of LS-URF-140 after burn-through testing in the test frame (left) and after being removed from the test frame (right).

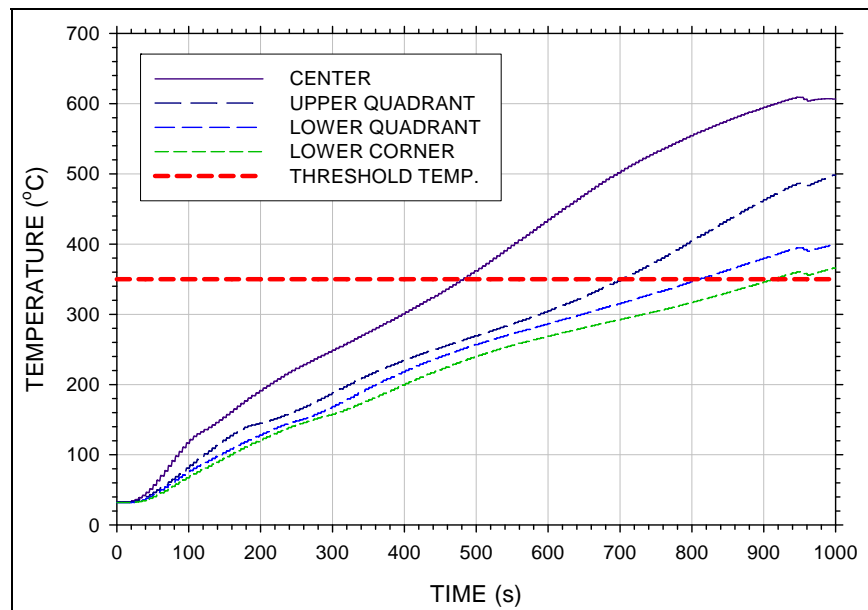


Figure 18. Unexposed surface temperatures measured during exposure of the PittChar XP/Polyurea coating.

As shown in figure 17, all backside temperatures did not exceed the temperature threshold until after 900 s (15 min) of exposure to the burner. Similar to that observed with the Nullifire System E coating, the intumescent material in this test remained in place over the duration of the exposure and after the panel was removed from the test frame. Posttest intumescent char thickness measurements resulted in a 21.3-mm average thickness. A photograph of the intumescent coating following the fire exposure is provided in figure 19.



Figure 19. Photograph of the PittChar XP/Polyurea coating after burn-through testing.

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## 14. Conclusions

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Burn-through tests were conducted on three coating samples in the vertical orientation with an exposure heat flux comparable to that expected from a UL 1709 exposure fire. Temperature data were collected in order to compare the insulating performance of the each coating sample. A plot of the centerline temperature profile on the unexposed side of each of the test panels is provided in figure 20 for comparison.

The combination of PittChar XP/Polyurea coating had the best insulating performance of all coatings tested, resulting in the longest times for the first and all temperatures to exceed the 350 °C threshold. However, the PittChar XP/Polyurea coating had the fastest time to ignition of any of three coatings tested. The Nullifire System E coating performed comparably to the PittChar XP/Polyurea coating, while being significantly more resistant to ignition. The light,

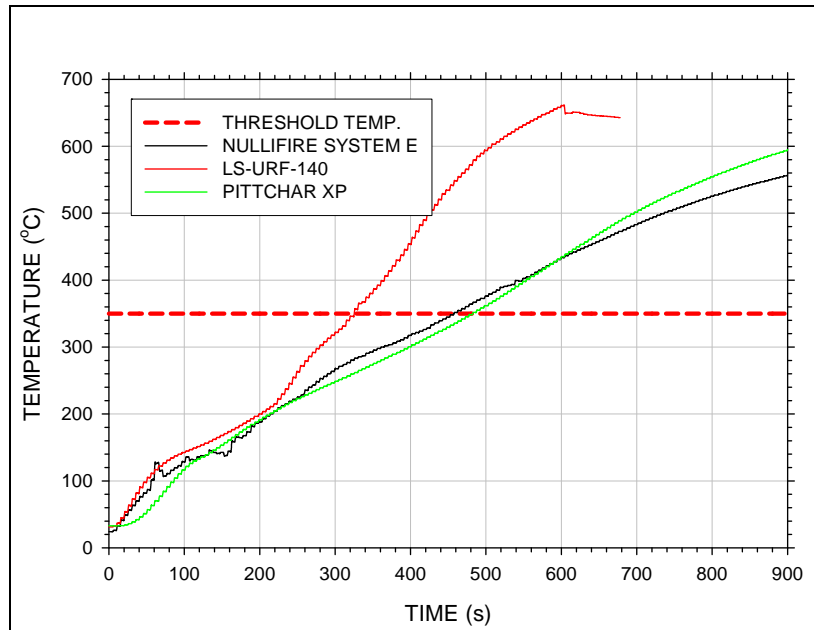


Figure 20. Comparison of insulating performance of intumescent coatings.

friable char produced by the LS-URF-140 coating was unable to remain in place during exposure to the propane burner, which may have contributed to the poor performance of the coating, with respect to providing an insulating barrier. Based upon the burn-through test results, the PittChar XP/Polyurea and the Nullifire System E should be further evaluated by applying these coatings to ammunition canisters and performing drop testing and furnace exposure testing on the coated canisters.

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## 15. References

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